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(54) METHOD FOR BONDING CERAMIC

(57)Abstract:

PURPOSE: To provide a highly reliable ceramic bonding member with a simple process by applying a slurry of the powder having the same composition as the powder constituting a ceramic compact on the surfaces of the compacts to be wetted and bonded, applying cold isostatic pressing to the compact and then sintering the compact.

CONSTITUTION: The surfaces of the ceramic compacts (uncalcined) to be bonded are brought into contact with an aq. binder soln., etc., and wetted. A slurry of the powder having the same composition as the compact is applied as a binder on the surfaces which are firmly held to each other. The assembly is then dried, subjected cold isostatic pressing and then sintered to bond the compacts. The bonded compacts are calcined to obtain a ceramic product. The base material and the bonding part are continuous in composition, and high homogeneity is secured in the product.

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CLAIMS

[Claim(s)]
[Claim 1] between the colds after contact the front face (the following, and a "plane of composition") which be going to join ceramic Plastic solids in the water solution of a binder etc., make it humid, apply the slurry containing the powder of the presentation same as cement as the configuration powder of the above-mentioned ceramic Plastic solid to the plane of composition of a damp or wet condition, stick planes of composition to it and dry -- dead water -- the junction approach of the ceramics characterize by to give pressurization press processing and subsequently to carry out sintering processing.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention relates to the junction approach of the ceramics.
[0002]
[Description of the Prior Art] Recently, application of the ceramics is variously tried as a configuration member of the equipment and the device by which thermal resistance, corrosion resistance, abrasion resistance, etc. are demanded. Although the member also has many which have a complicated configuration, many cases are difficult for really manufacturing this as elegance from points, such as a configuration, dimensional accuracy, etc. required of a member. Then, the parts which divide it into two or more parts, and have a simple configuration as an approach of manufacturing a member with a complicated configuration are manufactured, and the approach of making the member which has a predetermined configuration is proposed by joining combining these.
[0003]
[Problem(s) to be Solved by the Invention] As adhesives the conventional junction approach However, Si3 N4, SiO2, MgO, CaO, etc., or La 2O3, Y2 O3, aluminum 2O3, and SiO2 The low eutectic-point constituent of the plural systems made into a constituent is used. etc. -- It is what baking processing is performed [what] anew and produces junction after applying this to the front face of the parts manufactured as a burned product. Therefore, the zygote (ceramic product which has a request configuration) obtained The reinforcement of the joint and corrosion resistance are lower than that of a base material, and when the coefficients of thermal expansion of a joint and the base material section differ, there are problems -- it is easy to produce heat distortion and a crack -- at the time of real use, and it is lacking in the dependability as a structural member.
[0004] And by the junction approach, since the baking process for manufacturing the burned product (parts) which it is going to join, and the baking process for joining them are needed, **** of a process is not escaped. Moreover, repeating baking processing also becomes the cause which produces the grain growth (big-and-rough-izing of an organization) of a sintered compact, and degradation (fall of reinforcement, toughness, etc.) of the product quality accompanying it. Then, this invention tends to offer the new junction approach which can give the same material property substantially to a joint with a base material, and does not need repetitive implementation of a baking process.
[0005]
[Means for Solving the Problem and its Function] The front face to which the junction approach of the ceramics of this invention tends to join ceramic Plastic solids Hereafter, contact a "(plane of composition)" in the water solution of a binder etc., and it is made humid. between the colds after applying the slurry containing the configuration powder of the above-mentioned ceramic Plastic solid, the powder of the same presentation, and a binder to the plane of composition of a damp or wet condition, sticking planes of composition to it and drying -- dead water -- pressurization press processing is given and it is characterized by subsequently carrying out sintering processing.
[0006]
[Function] while pasting up the plane of composition of Plastic solids by using as cement the slurry which the junction approach of this invention makes a processing object a ceramic Plastic solid (it is baking at last), and contains the powder of the same presentation as the configuration powder of the Plastic solid -- baking processing -- between point ****** and the cold -- dead water -- an isostatic pressing press is performed. between the cold -- dead water -- under the uniform operation of high welding pressure by the isostatic pressing press, press fit restoration is carried out into the minute opening of each Plastic solid from a junction interface, and the cement containing the powder of the same presentation as a Plastic solid produces an anchor effect at coincidence. The discontinuity of the junction interface of Plastic solids disappears substantially by this, and it will be in the **** condition of whether they are really fabricated.

Therefore, the ceramic product obtained by carrying out baking processing of this does not have the presentation-discontinuity for a base material part and a joint, and the material property which has advanced homogeneity is secured.
[0007] Hereafter, the junction approach of this invention is explained in order of a process. the ceramic Plastic solid set as the object of the junction approach of this invention -- between a 1 shaft rubber press and the cold -- dead water -- it is fabricated by the well-known fabricating methods, such as an isostatic pressing press, extrusion molding, injection molding, and a slip casting, and the class of the shaping technique is not asked. According to this invention approach, the plane of composition of the ceramic Plastic solid which it is going to join first is made humid, and cement (slurry) is applied to the plane of composition of a damp or wet condition. A point ****** plane of composition is made humid by purging the pore of the plane-of-composition surface of a Plastic solid to spreading of cement for making the fluidity of the cement which needs the dispersion medium of the cement (slurry) applied there to prevent being quickly absorbed in a Plastic solid and join planes of composition hold.
[0008] When water or alcohols may be used as the humid liquid, and a ceramic Plastic solid mixes with binders (for example, acrylic resin binder etc.) as a shaping assistant and is fabricated by the configuration raw material powder, it is also desirable to use the water solution containing it and a binder of the same kind as humid liquid. Humid-ized processing of a plane of composition should just perform the part of a Plastic solid by being immersed in humid liquid.
[0009] The cement applied to the plane of composition of the Plastic solid made humid makes the water solution containing a proper dispersion medium, for example, water, or alcohols, the binder with which it was preferably mixed as a shaping assistant of the configuration raw material powder of a Plastic solid, and a binder of the same kind carry out distributed suspension of the powder of the same presentation as the configuration raw material powder of a Plastic solid, and is prepared as a slurry of the concentration (10 - 30 % of the weight of for example, dispersion media) suitable for spreading. What is necessary is for dip coating or brush coating just to perform spreading to a plane of composition. The immersion time amount in the case of being based on dip coating is good as 10 - 60 seconds, and, in brush coating, it is good to repeat spreading 2 to 3 times and to apply to homogeneity in the whole plane of composition.

[0010] Cement is applied to the plane of composition of a Plastic solid, planes of composition are stuck, and it dries. Desiccation is performed by the air drying by requiring suitable time amount (it being 5-24Hr, for example, although based also on the size of a Plastic solid). after desiccation and between the cold -- dead water -- an isostatic pressing press is given and press fit restoration of the cement of a junction interface is carried out into the minute opening of a Plastic solid according to a uniform operation of high welding pressure. between the cold -- dead water -- an isostatic pressing press -- welding-pressure about 1000 to 1500 Kgf/cm2 ** -- it carries out and is attained with the sufficient result by the processing for about 30 - 120 seconds. Thereby, a junction interface disappears substantially and, as for the joint of Plastic solids, a condition equivalent to a Plastic solid is really given.
[0011] between the above-mentioned colds -- dead water -- baking processing of a Plastic solid in which the isostatic pressing press was given -- an ordinary pressure sintering process (ambient pressure force about 10 Kgf/cm2 following) etc. -- applying -- a conventional method -- therefore, what is necessary is just to carry out
[0012]
[Example] A cylindrical ceramic Plastic solid and a disc-like ceramic Plastic solid are joined, and a flange pipe is manufactured.
The [I] ceramic (Plastic solid i) raw material powder kneading object nitriding silicon ceramics -- 100 weight sections sintering acid (a spinel, magnesia) -- Ten weight sections shaping assistant (an acrylic-acid methyl ester solution, 35% of concentration) -- The shaping 1 shaft rubber press fabricating method of 10 weight sections (ii) Plastic solid (however, welding-pressure:1500 Kgf/cm2, pressurization time amount:30 seconds) A cylindrical Plastic solid (outer diameter: 50mm, thickness:10mm, die-length:35mm) and a disc-like Plastic solid (outer diameter: 37mm, thickness:25mm) are fabricated. Relative density: 55%.
[0013] (Junction i) humid-ized processing humid liquid of the [II] Plastic solid: Acrylic-acid methyl ester solution (35% of concentration)
It is humid-ization by immersion (immersion time amount: 15 seconds) about the single-sided end face of a cylindrical Plastic solid and the single-sided face of a board of a disc-like Plastic solid used as a plane of composition.
(ii) -- spreading cement [of cement]: -- the slurry (10 % of the weight of dispersion mediums) which added the acrylic-acid methyl ester solution (35% of concentration) as a dispersion medium to the mixed powder (a nitriding silicon / sintering acid = 100/10, weight ratio) of the nitriding silicon ceramics and sintering acid (a spinel, magnesia). Cement (slurry) is applied twice to homogeneity in a brush in the plane of composition which the cylindrical Plastic solid and the disc-like Plastic solid made humid, and a spreading side is stuck, and it piles up up and down, and leaves and seasons naturally on a horizontal table. Drying time: 5Hr.

(iii) between the colds -- dead water -- the above-mentioned junction Plastic solid which carried out isostatic pressing press desiccation -- between the colds -- dead water -- the isostatic pressing press was given and processing for 1200 Kgf/cm2 x 30 seconds was performed.
(iv) -- between the baking processing colds -- dead water -- ordinary pressure sintering processing (0 ****: temperature, and nitrogen gas, one atmospheric-pressure, and time amount:1750-degree-Cx 10 minutes) was given after the isostatic pressing press, and the burned product of a collar-head pipe was obtained.
[0014] according to the line analysis by EPMA of the joint of the quality profit **** burned product of the [III] burned product, and its near base material section, a joint and the base material section show a numeric value with any almost same element, and it has the homogeneity continuity advanced in presentation. Moreover, deformation of the burned product is also very slight. As effectiveness that a joint and the base material section are homogeneous also in presentation, this is because the expansion and contraction for the joint produced at a baking process are substantially [as it of a base material] the same. The test piece for bend test (3x4x40, mm) was extracted, respectively from the base material section of the above-mentioned burned product, and the part containing a joint, by the tripartite bending method (span distance: 30mm), ordinary temperature and the disruptive strength in an elevated temperature (900 degrees C) were measured, and the result shown in Table 1 was obtained.
Table 1 Disruptive strength, Kgf/mm2 Ordinary temperature 900 degrees C A base material section test piece 68 56 Joint test piece 65 It is the homogeneity which is not different from the burned product of the Plastic solid which was substantially equivalent to the base material as for the joint, and was really fabricated from the comparison of the disruptive strength of the base material section and a joint shown in 55 table 1. It turns out that it has.
[0015]
[Effect of the Invention] The base material section and its joint do not have problems, such as deformation, a crack, etc. which has a continuity homogeneous to altitude also in presentation, and originates in a base material like a conventional method, the presentation-discontinuity of a joint, and it, the homogeneous material property of the burned product of a Plastic solid and coincidence is just really guaranteed, and, as for the burned product of the complicated configuration which joins the ceramic Plastic solid of a simple configuration by this invention approach, and is acquired, the burned product is rich in the dependability as a structural member. Moreover, according to the junction approach of this invention, the effectiveness which the conventional conjugation method which repeats a baking process, a **** intermediary, and baking can be managed with one process, and is acquired by simplification and energy saving of a process is also size.

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(54)【発明の名称】　セラミックスの接合方法

(57)【要約】

【構成】　セラミックス成形体同士の接合しようとする表面（接合面）を湿潤化し、湿潤状態の接合面に、セラミックス成形体と同じ組成の粉末とバインダを含有するスラリーを塗布して接合面同士を密着させ、乾燥した後、冷間静水加圧プレス処理に付し、ついで焼結処理する。

【効果】　焼成品は、母材部分と接合部分の組成的な不連続性もなく、一体成形体の焼成品と同等の均質性を有し、構造部材としての信頼性にすぐれている。焼成品同士を接合する方法と異なり、焼成処理は一回で済み、工程が簡素でコスト的にも有利である。

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【特許請求の範囲】

【請求項１】　セラミツクス成形体同士の接合しようとする表面（以下、「接合面」）を、バインダの水溶液等と接触させて湿潤化し、湿潤状態の接合面に、接合剤として上記セラミツクス成形体の構成粉末と同じ組成の粉末を含有するスラリーを塗布して接合面同士を密着させ、乾燥した後、冷間静水加圧プレス処理に付し、ついで焼結処理することを特徴とするセラミツクスの接合方法。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は、セラミツクスの接合方法に関する。

【０００２】

【従来の技術】近時、耐熱性、耐食性、耐摩耗性等が要求される装置・機器の構成部材としてセラミツクスの応用が種々試みられている。その部材には複雑形状を有するものも多いが、部材に要求される形状・寸法精度等の点から、これを一体品として製造することは多くの場合困難である。そこで、形状の複雑な部材を製造する方法として、それを複数の部分に分割して単純な形状を有するパーツを製作し、これらを組合せて接合することにより所定の形状を有する部材に仕上げる方法が提案されている。

【０００３】

【発明が解決しようとする課題】しかしながら、従来の接合方法は、接着剤として、Si_3N_4，SiO_2，MgO，CaO等、あるいはLa_2O_3，Y_2O_3，Al_2O_3，SiO_2等を構成成分とする多元系の低共融点組成物を使用し、これを焼成品として製作されたパーツの表面に塗布したうえ、あらためて焼成処理を施して接合を生じさせるものであり、従つて得られる接合体（所望形状を有するセラミツクス製品）は、その接合部の強度や耐食性が、母材のそれよりも低く、また接合部と母材部との熱膨張係数が異なることにより実使用時に熱歪みやクラツクを生じ易い等の問題があり、構造部材としての信頼性に乏しい。

【０００４】しかも、その接合方法では、接合しようとする焼成品（パーツ）を製作するための焼成工程と、それらを接合するための焼成工程とを必要とするため、工程の煩瑣を免れない。また焼成処理を反復することは、焼結体の結晶粒成長（組織の粗大化）と、それに伴う製品品質の劣化（強度、靭性等の低下）を生じる原因ともなる。そこで本発明は、接合部に母材と実質的に同一の材料特性を付与することができ、かつ焼成工程の反復実施を必要としない新規接合方法を提供しようとするものである。

【０００５】

【課題を解決するための手段および作用】本発明のセラミツクスの接合方法は、セラミツクス成形体同士の接合

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しようとする表面（以下、「接合面」）を、バインダの水溶液等と接触させて湿潤化し、湿潤状態の接合面に、上記セラミツクス成形体の構成粉末と同じ組成の粉末とバインダを含有するスラリーを塗布して接合面同士を密着させ、乾燥した後、冷間静水加圧プレス処理に付し、ついで焼結処理することを特徴としている。

【０００６】

【作用】本発明の接合方法は、セラミツクス成形体（未焼成である）を処理対象とし、かつその成形体の構成粉末と同一組成の粉末を含むスラリーを接合剤として成形体同士の接合面を接着すると共に、焼成処理に先立つて、冷間静水等方加圧プレスを行う。その冷間静水等方加圧プレスによる高加圧力の均一な作用下に、成形体と同一組成の粉末を含む接合剤が、接合界面から各成形体の微小空隙内に圧入充填され、同時にアンカー効果を生じる。これにより成形体同士の接合界面の不連続性が実質的に消失し、それらがあたかも一体成形されたものであるかの如き状態となる。従つて、これを焼成処理して得られるセラミツクス製品は、母材部分と接合部分との組成的な不連続性がなく、高度の均質性を有する材料特性が確保される。

【０００７】以下、本発明の接合方法について工程順に説明する。本発明の接合方法の対象となるセラミツクス成形体は、一軸ラバープレス、冷間静水等方加圧プレス、押出成形、射出成形、スリツプ・キヤスティング等、公知の成形法により成形されたものであり、成形手法の種類を問わない。本発明方法によれば、まず接合しようとするセラミツクス成形体の接合面を湿潤化し、湿潤状態の接合面に接合剤（スラリー）を塗布する。接合剤の塗布に先立つて接合面を湿潤化するのは、成形体の接合面表層の気孔をバージすることにより、そこに塗布される接合剤（スラリー）の分散媒が成形体中に急速に吸収されるのを防止し、接合面同士の接合を行なわせるに必要な接合剤の流動性を保持させるためである。

【０００８】その湿潤液として水、またはアルコール類等を使用してよく、またセラミツクス成形体が、その構成原料粉末に成形助剤としてバインダ（例えばアクリル樹脂バインダ等）を混和して成形されたものである場合は、それと同種のバインダを含む水溶液を湿潤液として使用するのも好ましいことである。接合面の湿潤化処理は、例えば成形体のその部分を湿潤液に浸漬することにより行えばよい。

【０００９】湿潤化した成形体の接合面に塗布される接合剤は、成形体の構成原料粉末と同一組成の粉末を、適宜の分散媒、例えば水、またはアルコール類、好ましくは成形体の構成原料粉末の成形助剤として混和されたバインダと同種のバインダを含む水溶液に分散懸濁させ、塗布に適した濃度（例えば分散媒１０～３０重量%）のスラリーとして調製される。接合面に対する塗布は、浸漬法、あるいは刷子塗り等により行えばよい。浸漬法に

よる場合の浸漬時間は例えば１０～６０秒としてよく、また刷子塗りの場合は、２～３回塗布を繰返し、接合面の全体に均一に塗布するのがよい。

【００１０】成形体の接合面に接合剤を塗布し、接合面同士を密着させて乾燥する。乾燥は、例えば自然乾燥により、適当時間（成形体のサイズにもよるが、例えば５～２４Ｈｒ）を要して行なわれる。乾燥の後、冷間静水等方加圧プレスに付し、高加圧力の均一な作用により、接合界面の接合剤を成形体の微小空隙内に圧入充填させる。その冷間静水等方加圧プレスは、加圧力約１０００～１５００Ｋｇｆ／ｃｍ² とし、約３０～１２０秒の処＊

＊理により首尾よく達成される。これにより、成形体同士の接合部は、接合境界面が実質的に消失し、一体成形体と同等の状態が与えられる。

【００１１】上記冷間静水等方加圧プレスが施された成形体の焼成処理は、常圧焼結法（雰囲気圧力　約１０Ｋｇｆ／ｃｍ² 以下）等を適用し常法に従つて行えばよい。

【００１２】

【実施例】円筒状セラミツクス成形体と円盤状セラミツクス成形体とを接合しフランジパイプを製作する。

［Ｉ］セラミツクス成形体

（ｉ）原料粉末混練物

窒化けい素セラミツクス　…１００重量部

焼結助剤（スピネル，マグネシア）　…１０重量部

成形助剤（アクリル酸メチルエステル溶液，濃度３５％）　…１０重量部

（ｉｉ）成形体の成形

一軸ラバープレス成形法（但し、加圧力：１５００Ｋｇｆ／ｃｍ² ，加圧時間：３０秒）により、円筒状成形体（外径：５０ｍｍ，肉厚：１０ｍｍ，長さ：３５ｍｍ）および円盤状成形体（外径：３７ｍｍ，厚さ：２５ｍｍ）を成形。相対密度：５５％。

【００１３】［ＩＩ］成形体の接合

（ｉ）湿潤化処理

湿潤液：アクリル酸メチルエステル溶液（濃度３５％）

接合面となる円筒状成形体の片側端面と円盤状成形体の片側盤面を浸漬（浸漬時間：１５秒）により湿潤化。

（ｉｉ）接合剤の塗布

接合剤：窒化けい素セラミツクスと焼結助剤（スピネル，マグネシア）の混合粉末（窒化けい素／焼結助剤＝１００／１０，重量比）に分散媒としてアクリル酸メチルエステル溶液（濃度３５％）を添加したスラリー（分散媒１０重量％）。円筒状成形体と円盤状成形体の湿潤化した接合面に接合剤（スラリー）を刷子にて２回均一に塗布し、塗布面を密着させて上下に重ね、水平台上に放置して自然乾燥。乾燥時間：５Ｈｒ。

（ｉｉｉ）冷間静水等方加圧プレス

※乾燥した上記接合成形体を、冷間静水等方加圧プレスに付し、１２００Ｋｇｆ／ｃｍ² ×３０秒の処理を施した。

（ｉｖ）焼成処理

冷間静水等方加圧プレスの後、常圧焼結処理（雰囲気：窒素ガス，１気圧，温度・時間：１７５０℃×１０分）に付し、フランジ付きパイプの焼成品を得た。

【００１４】［ＩＩＩ］焼成品の品質

得られた焼成品の接合部およびその近傍母材部のＥＰＭＡによる線分析によれば、接合部と母材部とはいずれの元素もほぼ同一の数値を示し、組成的に高度な均質連続性を有している。また、その焼成品の変形も極めて軽微である。これは接合部と母材部とが組成的にも均質であることの効果として、焼成工程で生じる接合部分の膨張・収縮が母材のそれと実質的に同一であるからである。

上記焼成品の母材部と、接合部を含む部分とからそれぞれ曲げ試験片（３×４×４０，ｍｍ）を採取し、三点曲げ法（スパン距離：３０ｍｍ）により、常温および高温（９００℃）での破壊強度を測定し、表１に示す結果を得た。

※

表　１

	破壊強度，Ｋｇｆ／ｍｍ²	
	常温	９００℃
母材部試験片	６８	５６
接合部試験片	６５	５５

表１に示した母材部と接合部の破壊強度の比較から、接合部は実質的に母材と同等で、一体成形された成形体の焼成品と変らない均質性を有していることがわかる。

【００１５】

【発明の効果】本発明方法により単純形状のセラミツクス成形体を接合して得られる複雑形状の焼成品は、母材部とその接合部とが組成的にも高度に均質な連続性を有し、従来法のような母材と接合部の組成的な不連続性とそれに起因する変形・クラツク等の問題もなく、その焼成品はあたかも一体成形体の焼成品と同時の均質な材料特性が保証され、構造部材としての信頼性に富むものである。また、本発明の接合方法によれば、焼成工程を反復する従来の接合法と異なつて、焼成は一工程で済み、工程の簡略化および省エネルギ化により得られる効果も大である。